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The Coordinated Development of Standards for Surface Chemical Analysis

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March 1985

Prepared for:

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

ABSTRACT

This report is based on a proposal to the Steering Committee of the Versailles Project on Advanced Materials and Standards (VAMAS) for the coordinated development amongst the VAMAS member states of standards for surface chemical analysis. VAMAS was established following the Economic Summit meeting at Versailles, France in 1982 that agreed on a number of projects relating to technology, growth, and employment. Specifically, VAMAS was organized to promote international coordination in the development of standards in a wide range of advanced material sectors. Surface chemical analysis was approved as a VAMAS Technical Working Area in June, 1984.

The report describes the growth and diversity of surface analysis in the development of advanced materials in modern technologies and, additionally, the use of surface analysis for improved films and coatings. The principal techniques of surface analysis in common use are identified and the technical limitations to accurate surface analyses identified. Accurate surface analyses require: (i) knowledge of the principles of the measurement method, (ii) knowledge of the behavior of the measuring instrument, and (iii) correct interpretation and expression of the final measured result. Standards for the measurement of surface composition with known accuracy and for the reliable determination of chemical state require: (a) a base of principles, definitions of terms, and suitable equations, (b) reference procedures for the measurement of intensities and spectral features, (c) procedures and data for instrument calibrations, (d) reference data for material parameters such as elemental sensitivity

factors, matrix terms, chemical state spectra, ion sputtering rates, etc; and (e) standard methods for specifying an analysis. Specific needs are identified for the common methods of surface analysis (Auger-electron spectroscopy, x-ray photoelectron spectroscopy, and secondary-ion mass spectroscopy) together with the needs for ion sputtering which is used to obtain composition versus depth information in films and coatings. Existing standards activities in the member countries of VAMAS are reviewed and suggestions are made for additional standards for surface chemical analysis.

Key words: reference data; reference materials; surface analysis; surface standards; Versailles Project on Advanced Materials and Standards

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1. The Versailles Project on Advanced Materials and Standards (VAMAS)

As a result of the initiatives originating in the 1982 Economic Summit meeting of Heads of State or Government at Versailles, there will be increased international coordination in the development of standards in a wide range of advanced materials sectors. This Economic Summit between the Heads of State or Government of Canada, France, the Federal Republic of Germany, Italy, Japan, the United Kingdom, and the United States of America and the Representatives of the European Communities, nominated a Working Group to report on the theme of collaborative projects relating to "Technology, Growth and Employment." Within this report, in the sector of "Improvement of Living Conditions, Employment and Protection of the Environment," we find the Versailles Project on Advanced Materials and Standards, otherwise known as VAMAS. Further information on similar Versailles projects has been published recently.¹

The Steering Committee of VAMAS, comprising representatives of all of the Member States listed above, including the Commission of the European Communities (CEC), has already considered and approved the launch of a small number of sector working parties with a further ten or so directly under review. The intention of the scheme is to stimulate the introduction of advanced materials into high technology products and engineering structures with the overall aim of encouraging international trade. This may be achieved through two routes: (i) by agreement between national experts responsible for setting national codes of practice and performance standards so that all such standards and codes are compatible and ISO standards are more easily established through the International

Organization for Standards; or (ii) through multilateral research aimed at furnishing the enabling scientific and metrological base necessary to achieve agreement on standards.

2. Significance of Surface Chemical Analysis

Increasingly today we see the introduction of sophisticated advanced materials configurations entering everyday use in thin film or surface layer form. This is integral to established technologies, such as microelectronics, as well as growing in importance in fields such as corrosion and catalysis. Coating technology, vacuum metallurgy, ion implantation, and plasma processing are but some of the many surface-science-related sectors which have acknowledged high growth potential for manufacturing industry. The development of very novel technologies in these areas depends on our ability to measure the chemical state of the films we deposit or the surfaces we prepare. Again, the reliability of these films or surfaces, and the assurances that we may make about them, depend on our ability to measure and verify surface compositions. The ubiquity of surface analysis through all sectors of industry is illustrated in the pie diagram based on United Kingdom manufacturing shown in Fig. 1; similar diversity has been documented in the U.S.A.²

The recognition of the importance of surfaces to technology may be seen in the growing number of special projects and fund allocations in this general area in the VAMAS Member States. The permeation of surface

analysis throughout the world is shown by the map in Fig. 2, although we should note that all of the surface analysis instrument manufacturers and about 90% of the research effort is concentrated within the Member States.

Because of the very newness of this area, the developments have outstripped the establishment of relevant standards. The problem has been recognized in the U.S.A. where the focus has been the E-42 Committee of the American Society for Testing and Materials (ASTM), and in Europe through the Community Reference Bureau. These programs have no formal relation with one another but attempt, as far as is practicable, to be complementary. Many other countries have projects which add to this body of work in an ad-hoc manner, as demonstrated at the third International Conference on Quantitative Surface Analysis at the U. K. National Physical Laboratory in November, 1983. International collaboration is urgently needed to make the most constructive use of this effort and to halt divergencies of approach. Until fuller cooperation is achieved, the prospect of surface chemical analyses entering into materials specifications and standards, in the way that bulk compositions do, cannot be envisaged.

3. Objective of the VAMAS Surface Chemical Analysis Working Party

A written proposal for the Coordinated Development of Standards for Surface Chemical Analysis was submitted by the present authors to the VAMAS Steering Committee and approved on June 18, 1984. The objective of this VAMAS activity is to produce, by coordinated effort, that body of reference procedures, data, and materials necessary to establish standards for surface chemical analysis.

4. Project Areas

We consider, here, work primarily, but not exclusively, associated with the established surface analysis techniques of Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), secondary ion mass spectroscopy (SIMS) and inert-gas ion sputtering for composition depth profiling (i.e., the measurement of composition versus depth). In this section we shall list the basic requirements. In section 5 we shall list current activities in the Member States and in section 6 we shall list some possible action areas in broad terms.

Each of the above techniques, in its ownway, gives information on how much of each element is present at the surface, the chemical state and spatial distribution, both across the surface and in depth through the surface layers. The accuracy with which these data may be determined with our current standards infra-structure leaves a great deal to be desired although, as has already been mentioned, some positive moves have already been initiated.

We may consider the structure of analysis using Fig. 3. Each analysis consists of three parts which must be understood by the analyst and which, if we are to establish a common basis, must be internationally agreed upon:

- (i) the principles of the physics of the measuring method
- (ii) the behavior of the measuring instrument
- (iii) the interpretation and expression of the measured result.

The basic principles of the physics of the techniques are vital since they lead to the working formulae which we shall use for quantitative analysis and which must be agreed when working to certain accuracies in given situations for each technique. These formulae involve the measurement of intensities by the instrument which are then related to the composition at the surface by functions of the material and the particular measurement process (which need to be tabulated) and by functions of the measuring instrument (which should be established by the user or be tabulated by the manufacturer and be established as time invariant). Additionally the measurements can give chemical information, the interpretation of which requires a similar hierarchy of understanding, data, and reference procedures. Therefore, we envisage that the analyst requires:

- (a) an agreed base of principles, definitions of terms and equations to relate the techniques to the measurement,
- (b) standards specifying how to measure intensities and spectral features - reference procedures,
- (c) procedures and data for calibrating instruments, determining instrument functions and inter-relating instruments of different designs,
- (d) reference data of elemental sensitivity factors, matrix terms, information depth, libraries of chemical state spectra, information on ion sputtering rates, radiation damage, etc.,
- (e) standard methods of specifying an analysis.

The above are required for all the surface analysis techniques, of which, in their order of popularity (i.e., number of instruments in use) and therefore priority we order AES, XPS and SIMS. Standards will also

need to be developed for additional techniques of surface analysis (e.g., ion-scattering spectroscopy, Rutherford backscattering spectroscopy, high-resolution electron energy-loss spectroscopy) as they become more prevalent. This we envisage as a future sector of work in this VAMAS project.

Below we list some of the most important problems currently requiring attention. This listing focuses attention on specific areas within (a) to (e) above and is in addition to those general comments. The list is not exhaustive but attempts to highlight the urgent problems.

4(a) Auger electron spectroscopy

- (i) Peak Area and Background Subtraction. In order to define the intensity to be measured in AES the electron transport in the solid should be understood as well as the decay process giving the peak structure. This enables the true background to be subtracted from the peak in the spectrum in order that the true peak area, and hence intensity, can be determined. In addition, practical but simpler equivalent background subtraction methods must be established and their accuracy evaluated to provide working rules for routine analysis.
- (ii) Differential Spectra. Peak intensities may alternatively be measured in the so-called differential mode. For analog (modulation) differentiation, an agreed specification is required for the way this is to be done in order that reference sensitivity factors may be used. For computer differentiation, an agreed method must again be adopted so that data will be transferable.

- (iii) Electron Inelastic Mean Free Path. An integral parameter of the electron transport in (i) is the electron attenuation length (AL) which is related to the inelastic mean free path (IMFP). The accuracy of theoretical expressions for the IMFP for pure elements and for atomic mixtures and compounds needs to be defined and the data base significantly increased. New methods of determining ALs and IMFPs need to be considered and rules given for establishing values for A and B in a material AB.
- (iv) Backscattering. Integral to the establishment of intensities from mixtures of phases and from compounds are what are commonly called matrix factors. These partly involve (iii) and partly the backscattering terms. The backscattering terms should be established for defined beam energies (say 2, 5 and 10 keV) and angles of incidence (0, 30 and 45°) with theoretical expressions and interpolation rules supported by a wide experimental data base covering metals, alloys, inorganic compounds and, if appropriate, organic compounds. Other contributions to matrix terms must be assessed.
- (v) Chemical State. Also stemming from (i) in the study of peak structure is the effect of chemical state. With the use of XPS instruments for AES this is now much more relevant and a data base of spectra (perhaps a library on floppy disc) should be compiled (see 4(b) (iv)).

- (vi) Relative Sensitivity Factors. A data bank of sensitivity factors for different elements and compounds is required for both the direct and derivative spectrum modes of intensity measurement. These are required for defined beam energies (say 2, 5 and 10 keV) and angles of incidence (0, 30 and 45°). If these are defined for one instrument, its intensity/energy response function (see viii) must be very accurately established.
- (vii) Formulae. Quantification can proceed from the above for a number of different situations such as monolayer partial coverage, microprecipitates, layered structures, etc. Each is calculated via a different basic equation involving different matrix terms. An agreed set of equations must be established in order that quantifications be readily understood.
- (viii) Spectrometer. In order to use universal data banks for quantification, the spectrometer intensity/energy function must be known. Reference methods, materials and data are required to calibrate spectrometer intensity and energy scales and to establish their constancy with time.
- (ix) Other instrumental parameters. For defining instrument performance and specifications a number of parameters must be measured by agreed methods, such as energy resolution, spatial resolution, and signal-to-noise ratio.
- (x) Ion sputtering. See under 4(d).

4(b) X-ray photoelectron spectroscopy

- (i) Peak Area and Background Subtraction. This problem is parallel to 4(a) (i).
- (ii) Electron Inelastic Mean Free Path. This problem is the same as 4(a) (iii).
- (iii) Matrix Terms. Matrix terms involving 4(b) (ii) have largely been ignored in XPS and require both experimental and theoretical consideration.
- (iv) Chemical State. This is very important and of much greater current use than 4(a) (v). Further data are required on spectra as a function of chemical state. Many data already exist in the literature but their quality is variable and the energy scales are not always properly referenced. These data need to be appraised and compiled initially as tabulated data for chemical shifts and later as spectra to show changes in satellite features. Both types of files should be in data banks to which new data could be readily added.
- (v) Relative Sensitivity Factors. A data bank of sensitivity factors for different elements and compounds is required for Al and Mg radiation. Effort needs to be focused on discrepancies between the theoretical and experimental data being produced. Angular asymmetry measurements are needed to enable the effects of different instrument configurations to be understood.
- (vi) Formulae. Here, again, many parallels with AES exist for requirements and methods.

(vii) Spectrometer. The spectrometer use and configuration in XPS can be more variable and complex than for AES. Considerably greater effort is therefore required to establish reference methods, data, and procedures to determine spectrometer intensity/energy response functions with known accuracy in order to achieve good quantification from the core data base.

(viii) Other Instrumental Parameters. See 4(a)(ix).

(ix) Ion Sputtering. See 4(d).

4(c) Secondary Ion Mass Spectroscopy

Here we must consider two forms of SIMS, static and dynamic. Static SIMS concerns surface analysis at the very low flux densities, before discernable ion bombardment damage occurs, whereas dynamic SIMS concerns thin film and bulk microscopic analysis generally for layers deeper than 10 nm from the surface. Modern instruments are beginning to bridge the gap but it is useful here to note that certain requirements are necessary for only one or other branch of the technique. Additionally, we should note that if an energetic neutral beam is used in place of the ion beam in static SIMS we have the technique called fast atom bombardment mass spectroscopy (FABMS). FABMS has great commonality with static SIMS but causes less damage and less sample charging.

(i) Intensities. Considerable effort is still required for both static and dynamic SIMS as well as FABMS to relate measured intensities to compositions theoretically. Matrix factors and cracking patterns are still poorly understood. Effort needs to be focused in this area but it will probably be some time before these techniques reach the state of certainty now

possible in AES and XPS (this is not meant to imply that AES and XPS are better than SIMS/FABMS since the latter provide far higher sensitivity than, and different data from the former).

- (ii) Chemical State. Cracking pattern spectra enable the precise bonding state of groups on surfaces to be identified in Static SIMS and FABMS. Libraries of spectra should be compiled (as tables or on floppy disc) for certain surfaces of common importance (e.g., organic coatings) to enable correct and quantitative interpretations to be made. The spectra should be for defined beams (Ar, Cs) at defined orientations and energies (1, 2, 5 keV), and for defined mass spectrometer take-off angles.
- (iii) Relative Sensitivity Factors. Because of the high matrix sensitivity these are not as universal as in XPS and AES. Nevertheless, since a considerable effort in both dynamic SIMS and static SIMS/FABMS is confined to a few matrices, some relative sensitivity factors should be compiled. This would involve bond groupings on organic surfaces in static SIMS/FABMS, on the one hand, and selected dopants in GaAs, Si or Ge in dynamic SIMS on the other. Additionally, to ensure traceability of the relative sensitivity factors, reference materials of appropriate composition require establishment and certification.
- (iv) Formulae. Parallels with AES exist for requirements and methods here as well as in XPS.

- (v) Spectrometer. To ensure correct instrument behavior and to use data banks and reference samples, the spectrometer ion energy discrimination, ion mass transmission, and ion detection functions must be established. Reference methods, data, and materials are required to calibrate the instruments and to ensure their constancy with time. The reference materials here may or may not be a selection from (iii) and may additionally characterize the energetic beam.
- (vi) Other Instrumental Parameters. See 4(a) (ix).
- (vii) Ion Sputtering. See 4(d).

4(d) Ion Sputtering

Ion or energetic neutral beams are involved with each of the above techniques as well as in others such as ion scattering spectroscopy (ISS). The following projects therefore relate to surface analysis in general. We need a better understanding of the ion-surface interaction leading to theoretical predictions and experimental data for the following.

- (i) Modification of the surface. For quantification we need to know how sputtering alters the composition of multi-element samples throughout the analysis depth. Predictions and data should be limited to standard conditions of beam energy, angle of incidence, and ion or neutral species.
- (ii) Modification of the material. For quantification we also need to know how the energetic ion alters the material beneath the surface region, the problem of atomic mixing, and the extent of differential sputtering (the amount by which one component is removed faster than another).

- (iii) Modification of the surface topography. This ill-understood topic is important for understanding both the depth resolution and variations in the depth profiling rate.
- (iv) Sputtering Rate. Essential to the quantification of the depth sputtered is the sputtering yield. Sputtering yields need to be tabulated for fixed conditions of ion energy, angle of incidence, and ion species for pure elements and, in addition, accurate rules for their use in multi-element samples should be established. Sputtering yields will alter with total fluence, with the development of surface topography and with temperature. These should be assessed at the same time.
- (v) Instrumental Parameters. Additionally, for the quantification of depth sputtered we need a reference material with known thickness, sputtering yield, and other parameters with a method for assessing and setting up the ion beam to obtain a reproducible current density. The latter requires a study of Faraday cup designs in order to establish a standard of practical usefulness and known accuracy.
- (vi) Formulae. Agreed formulae are required with an assessment of their accuracy for use by surface analysts.

5. Current Activities of a Standards, Measurement or Quantification Nature in Surface Chemical Analysis.

Below we list the current activity of organizations and individuals known to be involved in VAMAS-related work in surface chemical analysis.

5(a) U.S.A.

- (i) American Society for Testing and Materials (ASTM). The ASTM Committee E-42 on Surface Analysis was established in 1976 to advance the field of surface analysis and the quality of surface analyses. There are currently 10 active subcommittees dealing with separate aspects of surface analysis:

E42.01 Editorial

E42.02 Terminology

E42.03 Auger electron spectroscopy (AES)

E42.04 ESCA (or XPS)

E42.05 Ion scattering spectroscopy (ISS)

E42.06 Secondary ion mass spectroscopy (SIMS)

E42.07 Inactive

E42.08 Ion beam sputtering (IBS)

E42.09 Standard Reference Materials (SRM)

E42.10 Energetic Ion Analysis (EIA)

E42.11 Standard Reference Data (SRD)

Members of subcommittees consider work that is appropriate for standards in their respective areas and (i) consider and write standard definitions of terms (ii) consider, write and field test reference procedures for assessing the instruments and their components, (iii) consider, prepare and field test standard reference materials to characterize instrument performance, and (iv) consider and prepare algorithms for data analysis and identify needs for reference data. These activities lead to the sponsorship of round robins, as shown in Appendix

1. Standards already developed by the E-42 Committee and those under development are listed in Appendix 2. In addition, the Committee organizes symposia and workshops and arranges publications.

(ii) National Bureau of Standards (NBS)

The relevant work at NBS is conducted within the Surface Science Division. Division staff are active in ASTM topic areas with in-depth studies, preparation of Standard Reference Materials (SRMs), measurement of reference data for instrument calibration and for quantitative analysis, and in the publication of relevant review articles.

5(b) Europe

(i) Community Bureau of Reference (BCR)

The BCR assists individual projects in this area with work conducted in Member States. Current work covers AES, XPS, IBS and SIMS with the development of Certified Reference Materials (CRMs), Reference Methodology, and Reference Data. Round robins have been conducted, as shown in Appendix 3, leading to publication of work in the open literature and to CRMs.

(ii) U.K. National Physical Laboratory (NPL)

At NPL the relevant work is conducted within the Interfacial Microchemistry project of 3 to 4 staff members. Atomic standards are developed, CRMs prepared and Reference Methodology, Data and Procedures developed and tested through interlaboratory comparisons and round robins. Some are given in Appendix 3. The NPL organizes conferences on standards for surface analysis and publishes relevant review articles.

(iii) Societe Francaise du Vide (SFV)

The French Vacuum Society's Surface Science Group is involved in developing methods of spectrometer characterization, in standards preparation, in developing calculation codes, and in industrial applications of surface science.

5(c) International Union of Pure and Applied Chemistry (IUPAC)

The IUPAC Commission V.2 (microchemical techniques and trace analysis) has recently established a Working Group on Surface Analysis. This working group is chaired by Dr. M. Grasserbauer (Austria) and has representatives from France, the Federal Republic of Germany, South Africa, and the U.S.A. This group has developed plans to produce reference methods, reference data, and reference materials for specific problem areas in surface analysis.

6. Initial Projects Proposed For Coordination Through VAMAS

Scientists from all VAMAS Member States already contribute to our basic understanding of the analytical techniques. Whilst we wish to focus attention on certain objectives, as presented here, it must be recognized that some of the problems will not be solved in the immediate future. In order to promote improved analytical practices, the present authors plan to provide a review of all quantification procedures and basic equations so that the limits of accuracy for each is understood.

For AES, a coordinated effort on background subtraction methods for peak area analysis will be conducted in the U.S.A. Backscattering terms are a subject of intense analysis in Japan. Data are urgently required for electron attenuation lengths/inelastic mean free paths (IMFPs) in AES and XPS; the IMFP problem has been long recognized and requires a broad effort between many nations. Data banks for peak shapes as a function of chemical state may be considered as part of the parallel exercise for XPS and would fit well with current efforts at NBS. Likewise efforts to characterize electron spectrometer response function parallels that for XPS and fits well with current efforts at NPL.

For XPS background subtraction, studies to define peak areas could be coordinated between current efforts in the U.S.A., Federal Republic of Germany (F.R.G.), and U.K. The IMFP and spectrometer intensity-energy response functions are mentioned above. Chemical state data for peak shapes and positions are a current interest at NBS although it would be very useful to extend and broaden the information in the data bank. Matrix terms do not appear to be currently under investigation at all.

For static SIMS/FABMS, fragmentation patterns and standard spectra for certain organic surfaces are being compiled in the U.K. and data banks may be unified with that in the U.S.A. for XPS. Analysis of instrument intensity-mass-specie response functions is a current interest in the U.K. For dynamic SIMS, reference materials are required for the calibration of depth profiling of dopants in electronic materials. These are currently prepared in the ad-hoc manner in most Member States and coordinated effort here could be very productive.

For ion sputter depth profiling, reference materials are available or are becoming available from the NBS and NPL to define instrument operating conditions and sputtering rate. At NBS, the work on pure-element sputtering yields could be extended to meet the requirements detailed above and at NPL work on modifications of surface topography could be combined with the considerable efforts in the F.R.G. to understand the ion bombardment modifications of material compositions to provide a general framework in this sector.

Relative sensitivity factors are important for each of the techniques and at present only a skeletal data bank exists. A full program really needs to be pursued once the peak area and background methods described above, and other ancillary subjects, have been more firmly established. We would view this to be important as a second phase of work.

7. Implementation

The VAMAS Surface Chemical Analysis Working Party consists of the representatives from each of the member states as given in Table 1. These individuals have been asked to identify scientists in their countries who have expertise in the areas listed in section 4 and who could contribute to this VAMAS project. In addition, they have been asked to identify additional specific standards projects to supplement the listing in Section 4. They would also interact with the appropriate national standards body of their state.

It is expected that VAMAS-sponsored projects may be of several different types. First, such projects may involve work by one or more individuals in a particular nation on a given topic. Examples of such

projects may be found in current activities of the ASTM E-42 Committee on Surface Analysis in the USA; recent accounts of the work of this committee have been published by Nelson³ and Holloway.⁴ Second, projects may involve work on a given topic by scientists in two or more nations. Examples of such projects are interlaboratory comparisons of measurements of the same type, for example that published by Clegg et al.⁵ Third, projects may involve coordination with other appropriate national and international organizations, for example the International Union of Pure and Applied Chemistry, the Community Bureau of Reference of the C.E.C., the ASTM E-42 Committee, etc.

The principal objectives of this VAMAS activity in surface chemical analysis are (1) to ensure international coordination of standards-related activities before national standards are adopted, and (2) stimulate the development of needed standards on an international basis. Individuals who feel that their current or projected work would complement or add to this VAMAS project are invited to contact their national working party representative (Table 1) for further information and discussion. Individuals of countries not a signatory to the VAMAS agreement are also welcome and should contact a representative that they know or the Chairman (C. J. Powell).

The VAMAS Surface Chemical Analysis Working Party will meet at regular intervals to coordinate planned activities and to review progress. Such meetings are expected to be held annually. The first meeting is planned to be held at the time of the European Conference on Applications of Surface and Interface Analysis, Veldhoven, The Netherlands, October

14-18, 1985. A second meeting is expected at the time of the Sixth International Conference on Solid Surfaces and the Tenth International Vacuum Congress, Baltimore, U.S.A., October 27-31, 1986.

Appendix 1. Round Robins Sponsored by ASTM Committees E-42 on Surface
Analysis and D-32 on Catalysts

1. D-32 AES/XPS round robin on insulators and catalysts
T. E. Madey, C. D. Wagner and A. Joshi, J. Electron
Spectrosc. Relat. Phenom. 10, 359 (1977)
2. E-42.04 XPS round robin on gold and copper foils, C. J.
Powell, N. E. Erickson and T. E. Madey, J. Electron
Spectrosc. 17, 361 (1979)
3. E-42.03 AES round robin on gold and copper foils, C. J.
Powell, N. E. Erickson and T. E. Madey, J. Electron
Spectrosc. 25, 87 (1982)
4. E-42.08 Sputter rate determination for Ta₂O₅ on Ta, A. J.
Bevolo, Surf. Interface Anal. 3, 240 (1981)
5. E-42.03 AES round robin on Au/Cu binary alloys (in process)
Contact: P. H. Holloway, University of Florida, U.S.A.
6. E-43.05 ISS round robin on Ag/Cu/Au ternary alloys (in
process)
Contact: A. Miller, Alcoa Laboratories, U.S.A.
7. E-42.06 SIMS round robin on ¹¹B implanted in Si (in process)
Contact: R. J. Colton, Naval Research Laboratory, U.S.A.
8. E-42.08 Sputter rate round robin for SiO₂ on Si (in process)
Contact: A. J. Bevolo, Ames Laboratory, U.S.A.
9. E-42.09 Standard reference material characterization - Ni/Cr
layered structures (in process)
Contact: J. Fine, National Bureau of Standards, U.S.A.

10. E-42.10 Standard reference materials for high-energy ion
techniques - Au/Ti layered structures (in process)

Contact: J. A. Borders, Sandia National Laboratories, U.S.A.

11. E-42.04 Comparison of core-level binding energies determined
by x-ray photoelectron spectroscopy with instruments
calibrated using a field-emission electron source (in
process).

Contact: C. R. Anderson, Naval Surface Weapons Center, U.S.A.

Appendix 2. Standards and Recommended Practices Produced by ASTM

Committee E-42 on Surface Analysis

(a) Published Standards

- E673-84: Standard Definitions of Terms Relating to Surface Analysis.
- E684-83: Practice for the Approximate Determination of the Current Density of Large-Diameter Ion Beams for Depth Profiling of Surfaces.
- E827-83: Standard Practice for Elemental Identification by Auger Electron Spectroscopy
- E902-83: Standard Practice for Checking the Operating Characteristics of X-Ray Photoelectron Spectrometers
- E983-84: Standard Guide for Electron Beam Effects in Auger Electron Spectroscopy
- E984-84: Standard Guide for Identifying Chemical Effects and Matrix Effects in Auger Electron Spectroscopy
- E995-84: Standard Guide for Background Subtraction Techniques in Auger Electron Spectroscopy
- E996-84: Standard Practice for Reporting Data in Auger Electron Spectroscopy
- E1015-84: Standard Practice for Reporting Spectra in ESCA
- E1016-84: Standard Practice for Determining and Specifying the Properties of Electrostatic Electron Spectrometers

(b) Standards Currently being Balloted

Additions to Standard Definitions of Terms Relating to Surface
Analysis (for ESCA and Energetic Ion Analysis methods)

Standard Guide for Specimen Handling in Auger Electron Spectroscopy
and X-Ray Photoelectron Spectroscopy

Standard Practice for Reporting Sputter Depth Profile Data in
Secondary Ion Mass Spectrometry (SIMS)

Standard Guide to Depth Profiling in Auger Electron Spectroscopy

Appendix 3. Reference Data Procedures and Round Robins Sponsored by the
NPL-BCR Program

1. XPS - Energy Calibration of Electron Spectrometers

- 1 - An Absolute Traceable Energy Calibration and the Provision of Atomic Reference Line Energies by M. T. Anthony and M. P. Seah, Surf. Interface Anal. 6, 95 (1984).
- 2 - Results of an Interlaboratory Comparison by M. T. Anthony and M. P. Seah, Surf. Interface Anal. 6, 107 (1984).

2. Quantitative XPS: The Calibration of Spectrometer Intensity - Energy Response Functions

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Figure Captions

- Fig. 1. The intensity of application of surface analysis, illustrated by the manufacturing sectors of the United Kingdom.
- Fig. 2. Countries of the world applying surface analysis.
- Fig. 3. The interacting components requiring specification to produce quantitative analyses and statements of chemical state, as illustrated schematically by components for x-ray photoelectron spectroscopy.

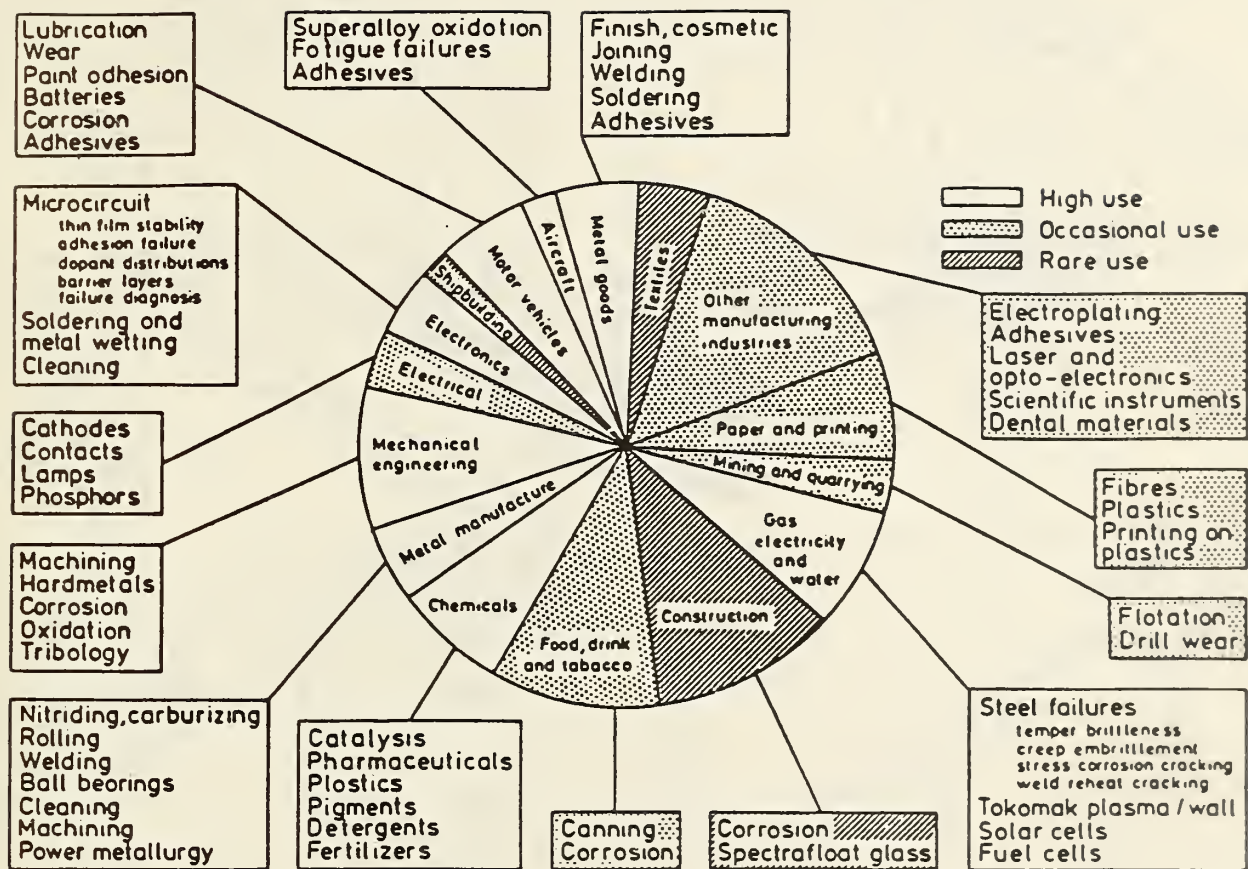


Fig. 1.

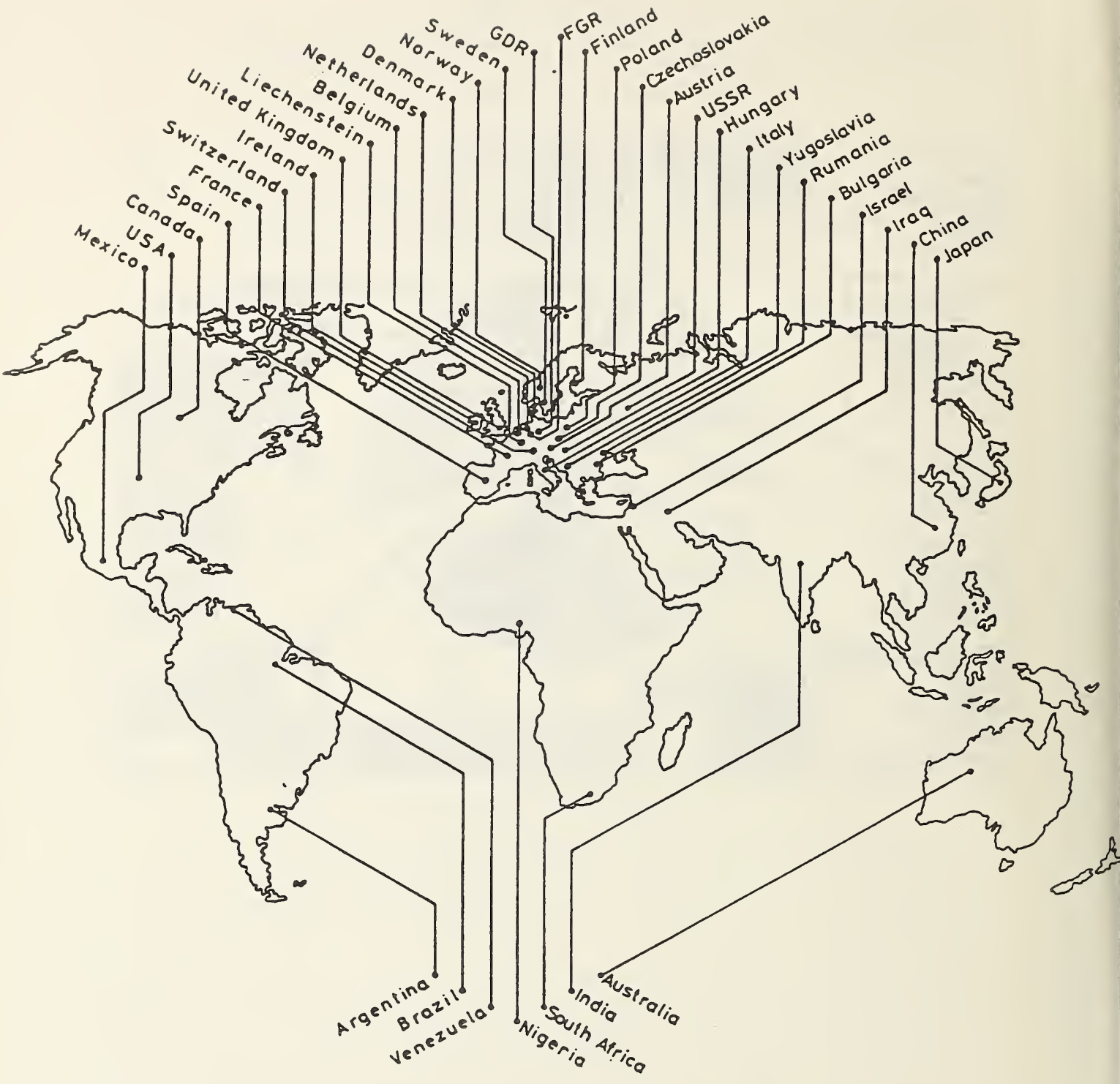


Fig. 2.

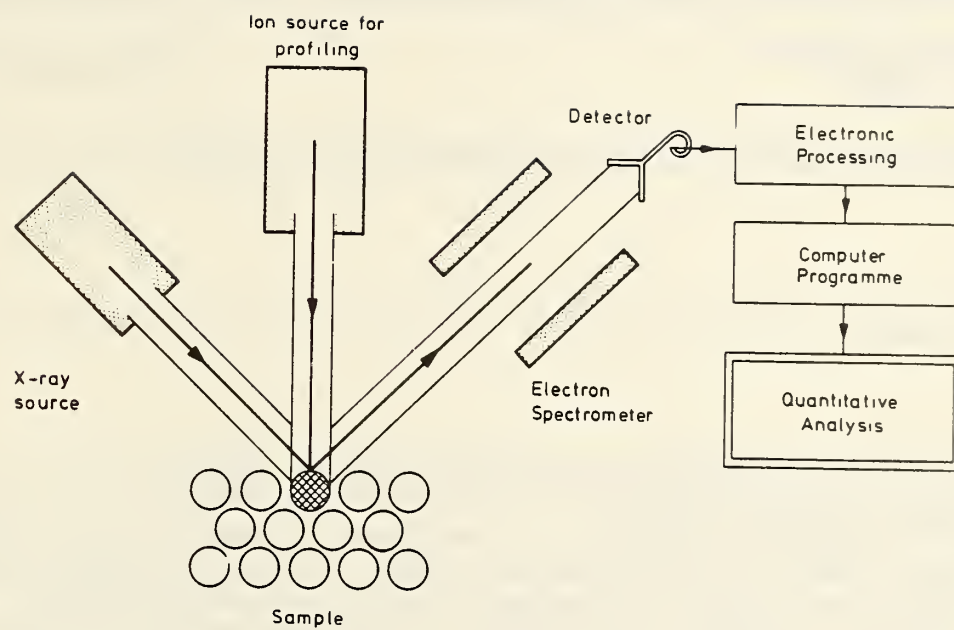


Fig. 3.

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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) This report is based on a proposal to the Steering Committee of the Versailles Project on Advanced Materials and Standards (VAMAS) for the coordinated development amongst the VAMAS member states of standards for surface chemical analysis. VAMAS was established following a meeting of the Heads of State or government at Versailles, France in 1982 that agreed on a number of projects relating to technology, growth, and employment. Specifically, VAMAS was organized to promote international coordination in the development of standards in a wide range of advanced material sectors. Surface chemical analysis was approved as a VAMAS Technical Working Area in June, 1984. The report describes the growth and diversity of surface analysis in the development of advanced materials in modern technologies and additionally, the use of surface analysis for improved films and coatings. The principal techniques of surface analysis in common use are identified and the technical limitations to accurate surface analyses identified. Accurate surface analyses require: (i) knowledge of the principles of the measurement method, (ii) knowledge of the behavior of the measuring instrument, and (iii) correct interpretation and expression of the final measured result. Standards for the measurement of surface composition with known accuracy and for the reliable determination of chemical state require: (a) a base of principles, definitions of terms, and suitable equations, (b) reference procedures for the measurement of intensities and spectral features, (c) procedures and data for instrument calibrations,			
12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) reference data; reference materials; surface analysis; surface standards; Versailles Project on Advanced Materials and Standards			
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(d) reference data for material parameters such as elemental sensitivity factors, matrix terms, chemical state spectra, ion sputtering rates, etc; and (e) standard methods for specifying an analysis. Specific needs are identified for the common methods of surface analysis, Auger-electron spectroscopy, x-ray photoelectron spectroscopy, and secondary-ion mass spectroscopy together with the needs for ion sputtering which is used to obtain composition versus depth information in films and coatings. Existing standards activities in the member countries of VAMAS are reviewed and suggestions are made for additional standards for surface chemical analysis.

